Engineering, Test & Technology
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Testing and Validation of High Temperature Materials

Department of Energy – ARPA-E
Ultrahigh-Temperature Materials for Energy Applications Workshop
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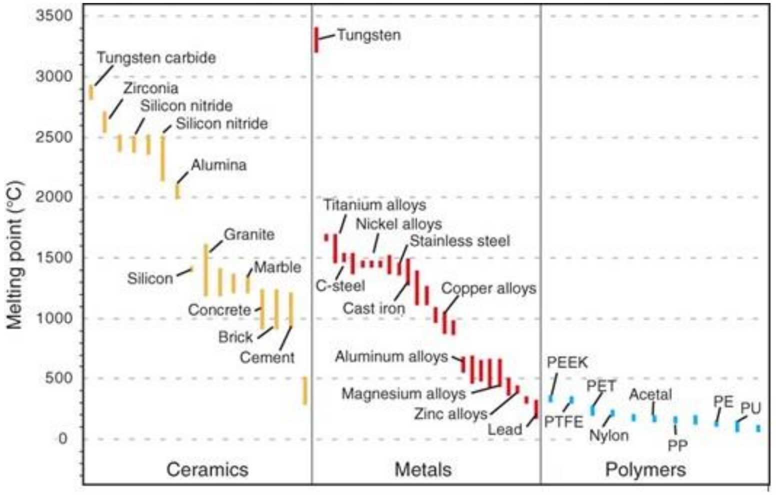
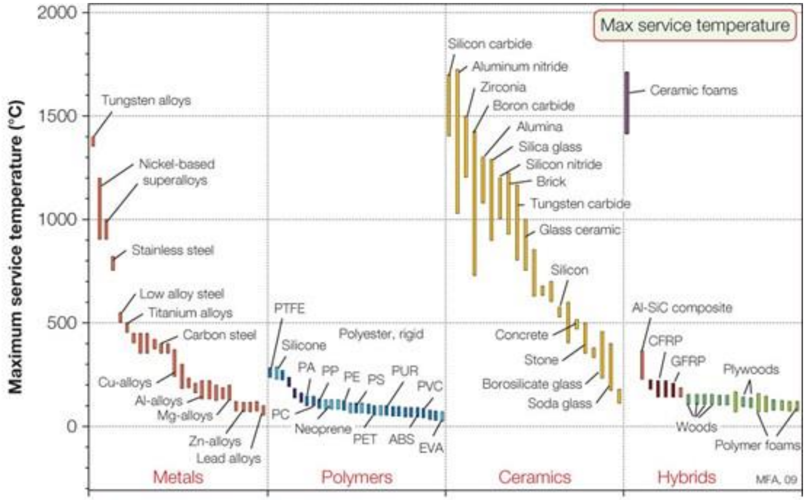
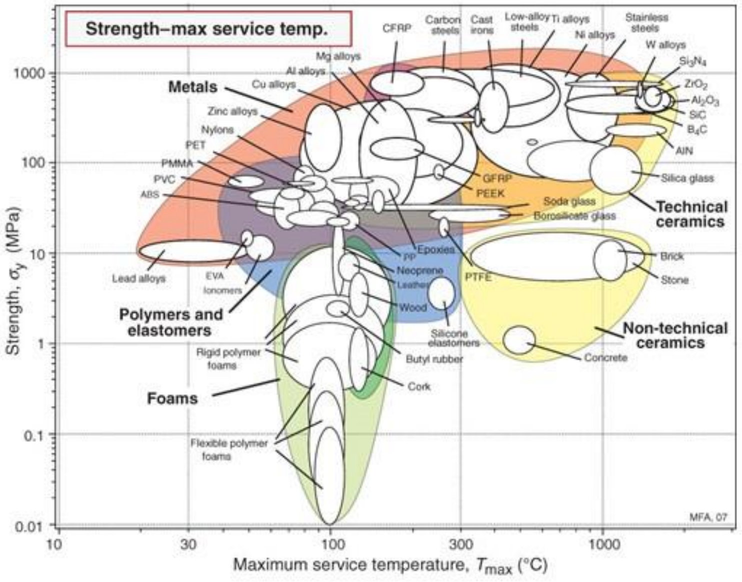
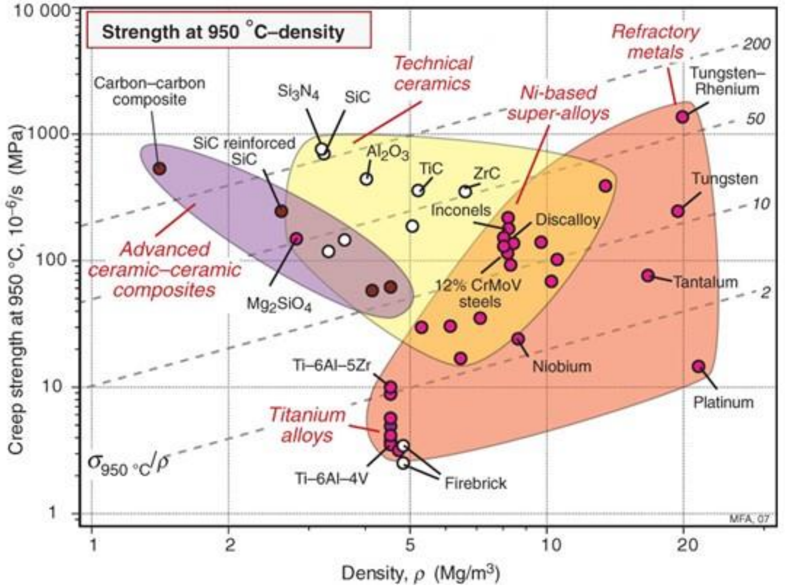
What is Maximum Service Temperature?

T °C	Materials	Applications	T K
1200 1000	Refractory metals: Mo, W, Ta Alloys of Nb, Mo, W, Ta Ceramics: Oxides Al_2O_3, MgO, etc. Nitrides, Si_3N_4, Carbides, SiC	Rocket nozzles Special furnaces Experimental turbines	1400
800	Austenitic stainless steels Nichromes, nimonics Nickel based super-alloys Cobalt based super-alloys Iron based super-alloys	Gas turbines Chemical engineering Petrochemical reactors Furnace components Nuclear construction	1200
600	Iron-based super-alloys Ferritic stainless steels Austenitic stainless steels Inconels and nimonics	Steam turbines Superheaters Heat exchangers	1000
400	Low-alloy steels Titanium alloys (up to 450 °C) Inconels and nimonics	Heat exchangers Steam turbines Gas turbine compressors	800
200	Fibre-reinforced polymers Copper alloys (up to 400 °C) Nickel, monels and nickel-silvers PEEK, PEK, PI, PPD, PTFE and PES (up to 250 °C)	Food processing Automotive (engine)	600
0	Most polymers (max temp: 60 to 150 °C) Magnesium alloys (up to 150 °C) Aluminum alloys (up to 150 °C) Monels and steels	Civil construction Household appliances Automotive Aerospace	400
−200	Austenitic stainless steels Aluminum alloys	Rocket casings, pipework, etc. Liquid O_2 or N_2 equipment	200
−273	Copper alloys Niobium alloys	Superconduction	0



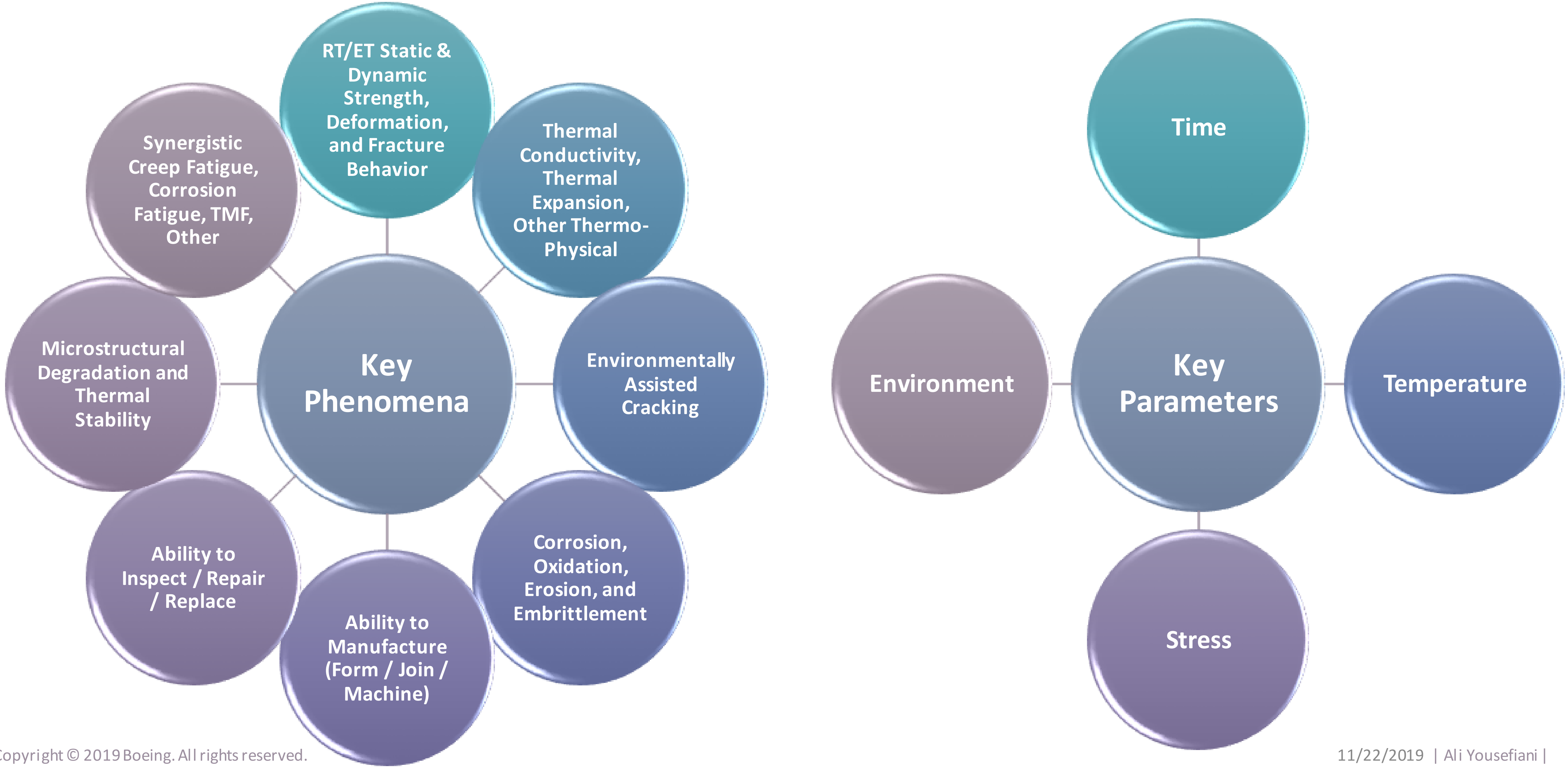




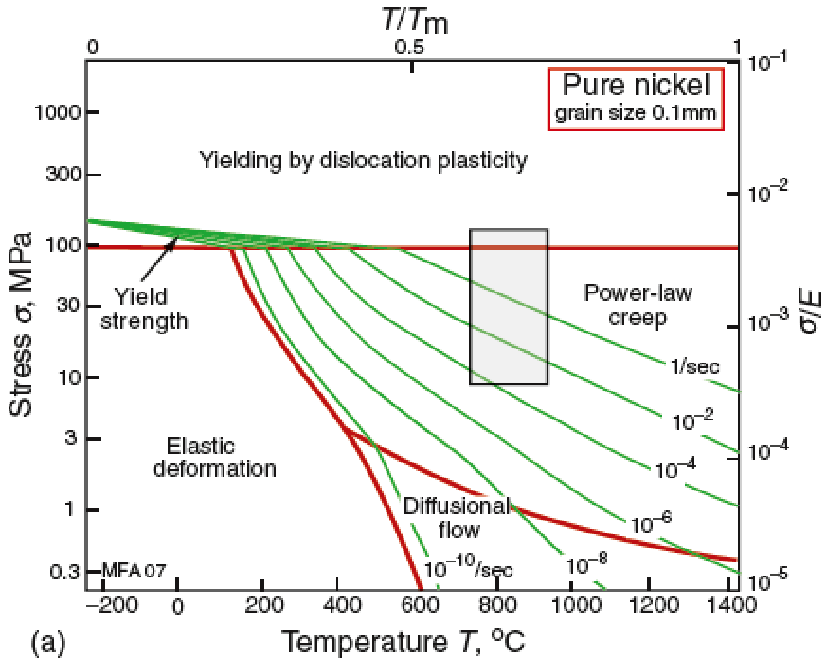


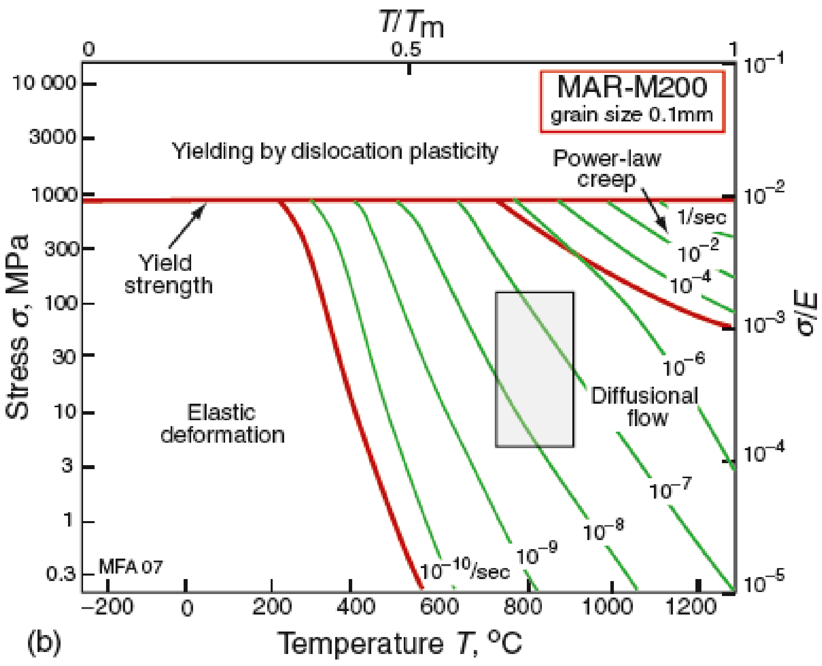
Materials: engineering, science, processing and design, 2nd edition, 2010, Michael Ashby, Hugh Shercliff, David Cebon

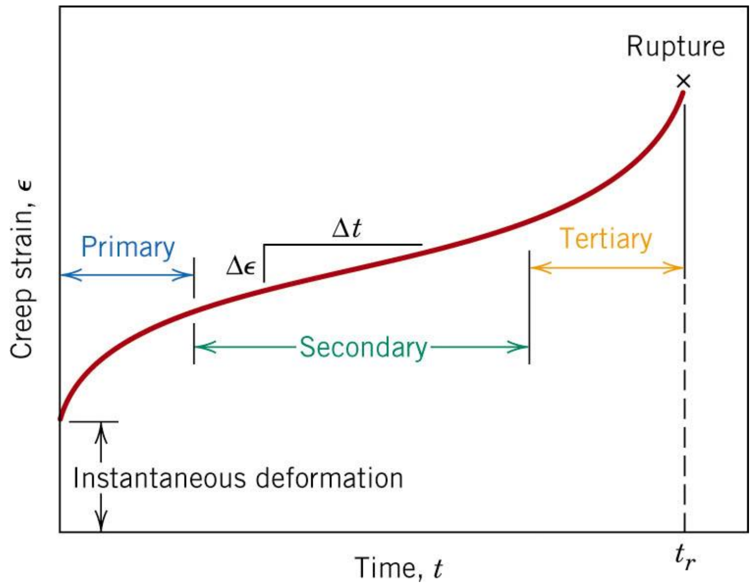
Key Phenomena/Parameters of Concern in Extreme Environments



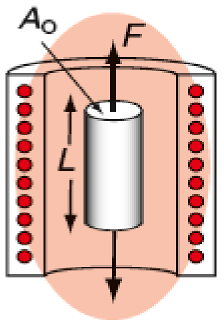
Deformation Mechanisms

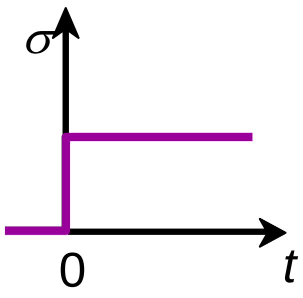






- Testing and Validation:
  - Quasi-static: MMPDS S/A/B Basis Design Allowables
  - Dynamic (time dependent): Bona Fide Average + 3/4/5 X Life





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Multiaxial Representative Stress Parameters

- Uniaxial stress conditions have commonly been employed in studies concerning failure of engineering materials at high temperatures.
- It has been observed that for uniaxial creep conditions a fundamental power law exists which simply relates the time to rupture, t_f, and the applied stress, σ, as follows (M and χ are stress independent constants for a given material and testing condition) :

$$t_f = M\sigma^{-\chi}$$

- In high temperature applications, however, the majority of the components are subject to stress states varying in both time and position. Under such complex loading conditions, the stress used in this equation must be modified to correctly predict rupture time.

$\sigma_{rep} = \alpha\sigma_1 + (1-\alpha)\sigma_e$	• Continuum mechanics approach. • σ_1 and σ_e contribute independently and represent the driving force for diffusional cavitation and creep deformation processes, respectively. • σ_e is the effective stress and α is a constant.
$\sigma_{rep} = \sigma_1^{\frac{\nu}{\chi}}\sigma_e^{\frac{\chi-\nu}{\chi}}$	• Continuum mechanics approach. • Contributions of different processes to creep rupture, driven by σ_1 and σ_e, are considered to be interdependent. • ν is a constant.

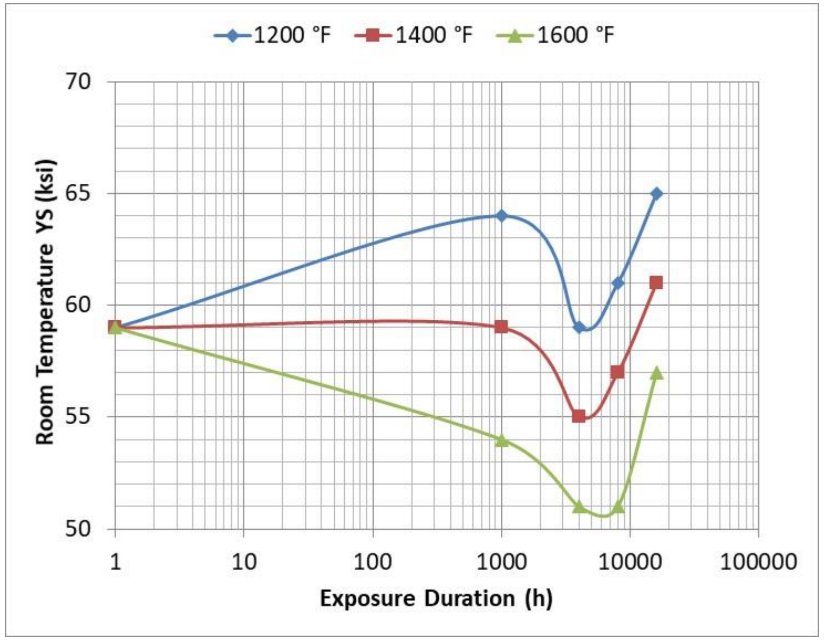
$\sigma_{rep} = \sigma_1$	• Based on early multiaxial creep rupture studies. • Applied under the condition of the progressive development of a homogeneous distribution of cavities at a level microscopically visible from the onset of testing.
$\sigma_{rep} = \sigma_e = \sqrt{\frac{(\sigma_1-\sigma_2)^2 + (\sigma_2-\sigma_3)^2 + (\sigma_1-\sigma_3)^2}{2}}$	• Based on early multiaxial creep rupture studies. • Applied under the condition that no significant cavitation occurs in samples other than that observed in the close vicinity of the rupture surface.
$\sigma_{rep} = \sigma_C = \sigma_1\sigma_e^{n-1}$	• Based on a cavity growth model developed by Rice. • Utilized for conditions where grain boundary cavitation is constrained by continuum creep rate of the surroundings. • n is the creep exponent.
$\sigma_{rep} = \sigma_F = 2.24\sigma_1 - 0.62(\sigma_2+\sigma_3)$	• Applicable for situations where cavitation is coupled with highly localized deformation processes, such as grain boundary sliding.

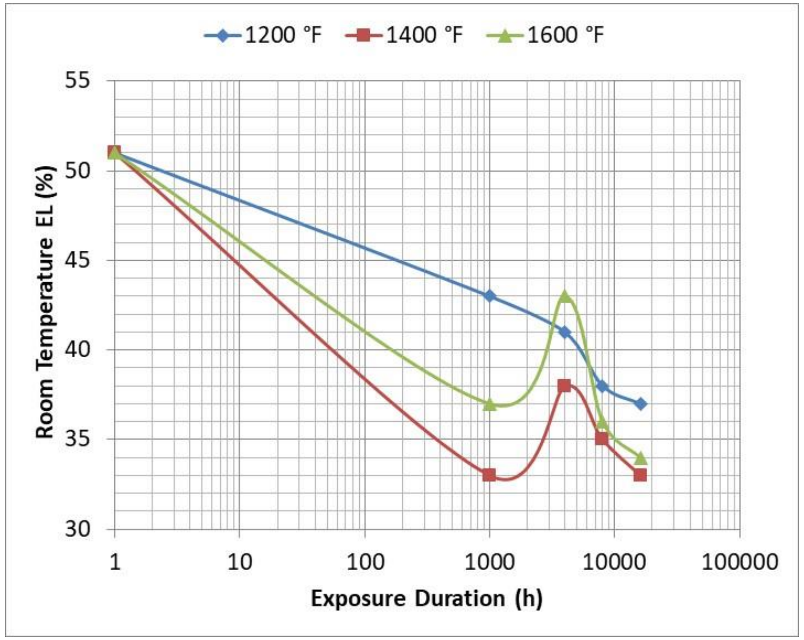
Thermal Fatigue and Creep fatigue

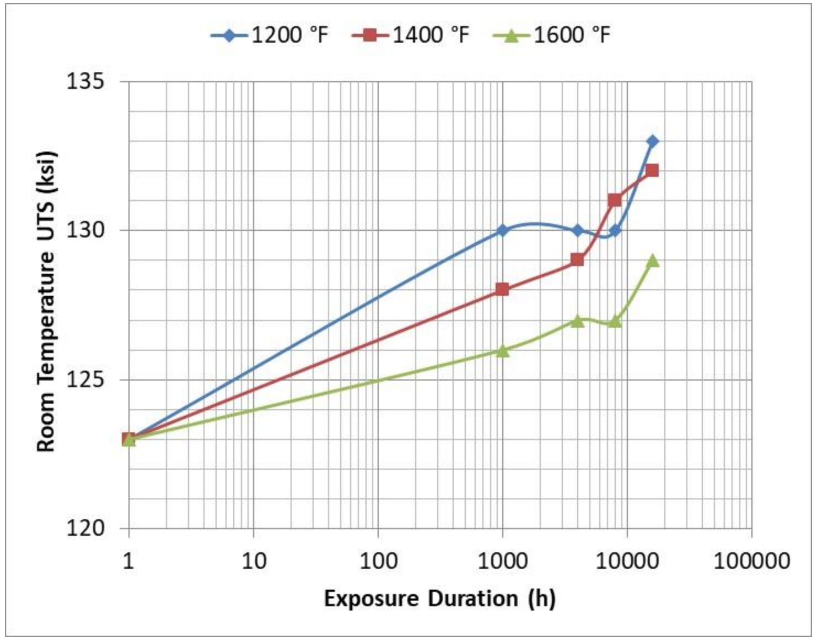
- Creep is not the only source of strain in high-temperature applications. Transient thermal gradients within a component can induce plastic strains; if these thermal gradients are applied repeatedly, the resulting cyclic strain can induce failure (thermal fatigue).
- Thermal fatigue has traditionally been treated similar to isothermal low cycle fatigue (LCF) at the thermal cycle maximum temperature. However, it is possible to analyze complex thermal cycles and to conduct thermomechanical fatigue (TMF) tests under controlled conditions.
- Thermomechanical and combined creep-fatigue loads can substantially decrease life at elevated temperatures as compared with that anticipated in simple creep loading or isothermal LCF tests.
- These damage mechanisms may act independently or in combination depending on materials and operating conditions, such as maximum and minimum temperatures, temperature range, mechanical strain range, strain rate, the phasing of temperature and strain, dwell time, or environmental factors.
- Different locations behave differently

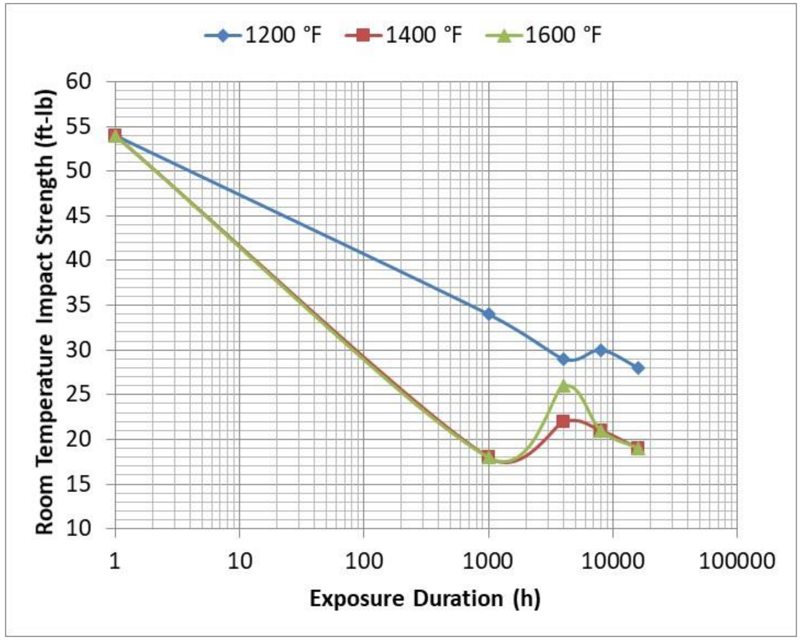
Thermal Stability

- Sometimes overlooked
- Life management of operating components in extreme environments should take into account the material is progressively degraded or damaged as operating time accumulates
- Damage is mainly in the form of changes in precipitate size and/or morphology that may result in softening (overaging) and reduced strength, or embrittlement and reduced resistance to fracture
- Temperatures of concern:
  - Incipient melting
  - Solutionizing
  - Annealing
  - Aging
  - Transformation
  - Etc



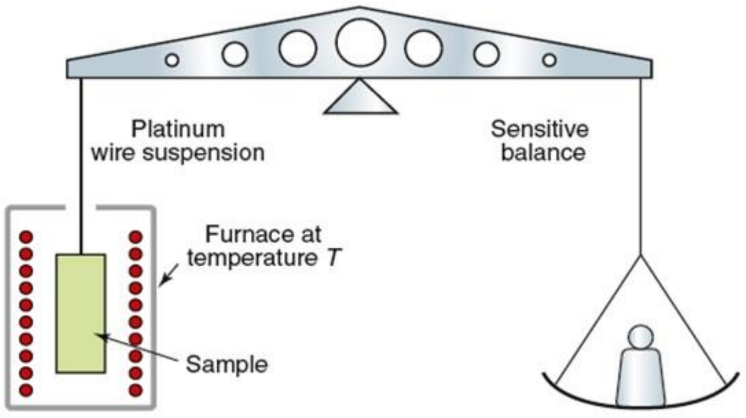


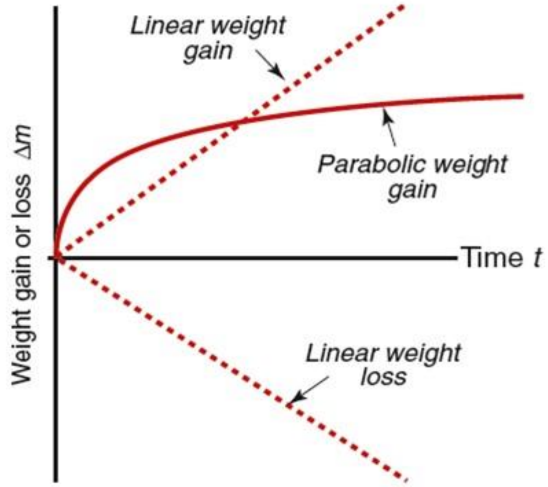




Forms of Corrosion

- While corrosion can take many forms, it is most generally defined as a chemical or electrochemical reaction between a material and its environment that produces a deterioration (change) of the material and its properties.
- A broad view would separate corrosion into two categories:
    - Not influenced by any other process
        - Uniform Corrosion
        - Localized Corrosion
        - Metallurgically Influenced Corrosion
    - Influenced by another process, such as the presence of stresses or erosion.
        - Mechanically Assisted Degradation
        - Environmentally Induced Cracking
- High Temperature Corrosion





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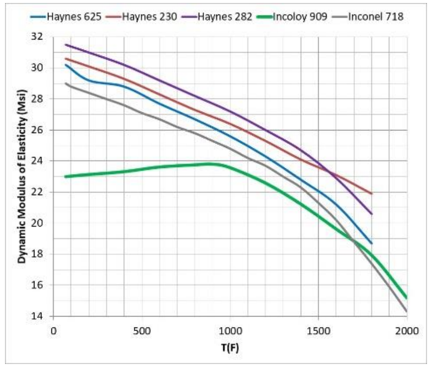
High-Temperature Corrosion

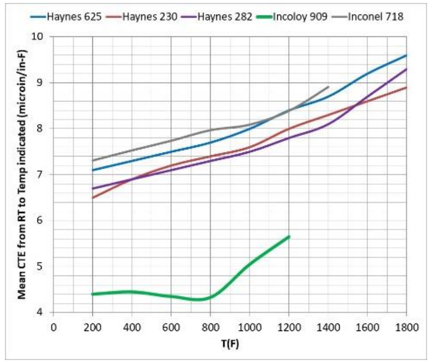
- Potential degradation mechanisms for HT corrosion:
  - Oxidation
  - Carburization and metal dusting
  - Sulfidation
  - Hot corrosion
  - Chloridation
  - Hydrogen interactions
  - Molten metals
  - Molten salts
  - Aging reactions, such as sensitization
  - Environmental cracking (stress-corrosion cracking and corrosion fatigue)
- Presence of molten salts or metals may induce other mechanisms, such as galvanic corrosion, crevice corrosion, and pitting corrosion.
- Impingement by solid particles may contribute to erosion-corrosion or accelerate corrosion in the various gaseous or molten environments.

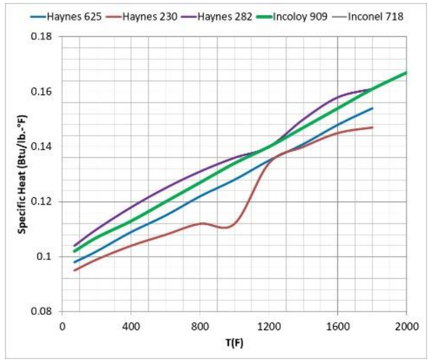
Environmentally Assisted Cracking (EAC)

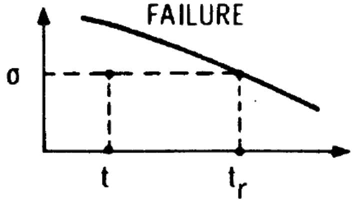
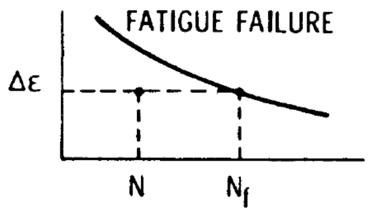
- Mechanisms of corrosion that induce cracking of materials as a result of exposure to their environment. This cracking may take the form of relatively slow, stable crack extension with a predictable growth rate or, as is often the case, unpredictable catastrophic fracture.
- Environmentally Assisted Cracking (EAC):
  - Stress-corrosion cracking (SCC)
  - Hydrogen damage (frequently referred to as hydrogen embrittlement - HE)
  - Liquid metal induced embrittlement (LMIE)
  - Solid metal induced embrittlement (SMIE)
- All of these phenomena generally are dependent on yield strength and applied stress. As both of these factors increase, resistance to EAC decreases. However, many differences between the various forms of environmentally induced cracking are encountered.

Design Space

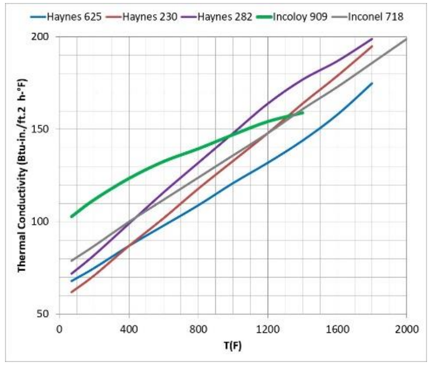


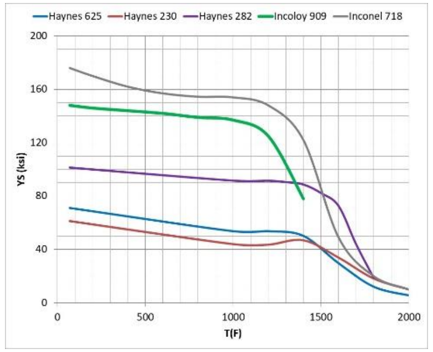


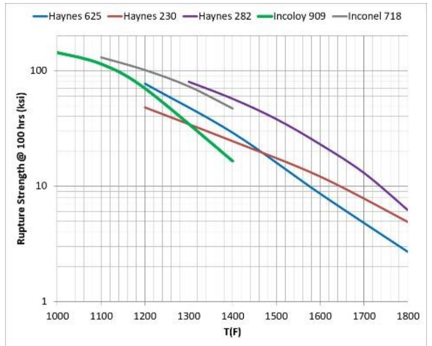


FAILURE MODE	STRESS-TIME-DEPENDENT	STRAIN-CYCLE-DEPENDENT
FAILURE CRITERION	CREEP RUPTURE FAILURE 	FATIGUE FAILURE 
MEASURE OF DAMAGE	LIFE FRACTION t/t_r	LIFE FRACTION N/N_f
ACCUMULATION OF DAMAGE	LINEAR SUMMATION TO 1 $\sum \frac{t}{t_r} = 1$ (AT FAILURE)	LINEAR SUMMATION TO 1 $\sum \frac{N}{N_f} = 1$ (AT FAILURE)
COMBINATION	$\sum \frac{t}{t_r} + \sum \frac{N}{N_f} = 1$ (AT FAILURE)	

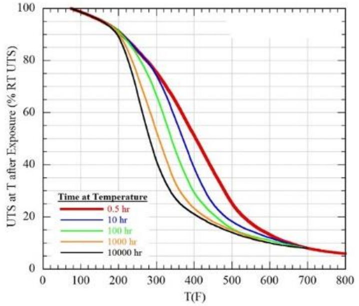
Exemplary Lifetime/Damage Assessment Methodology

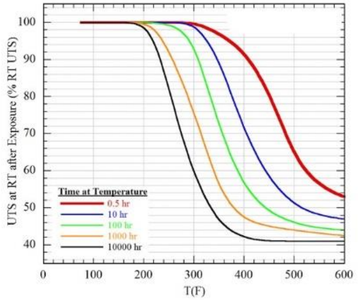






Synergistic Creep Fatigue, Corrosion Fatigue, TMF, Other

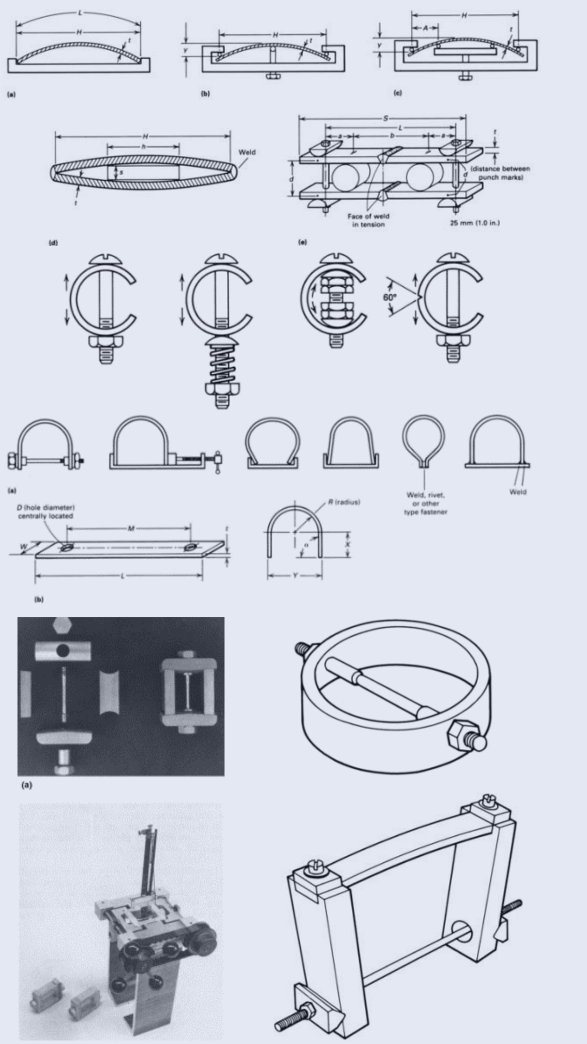




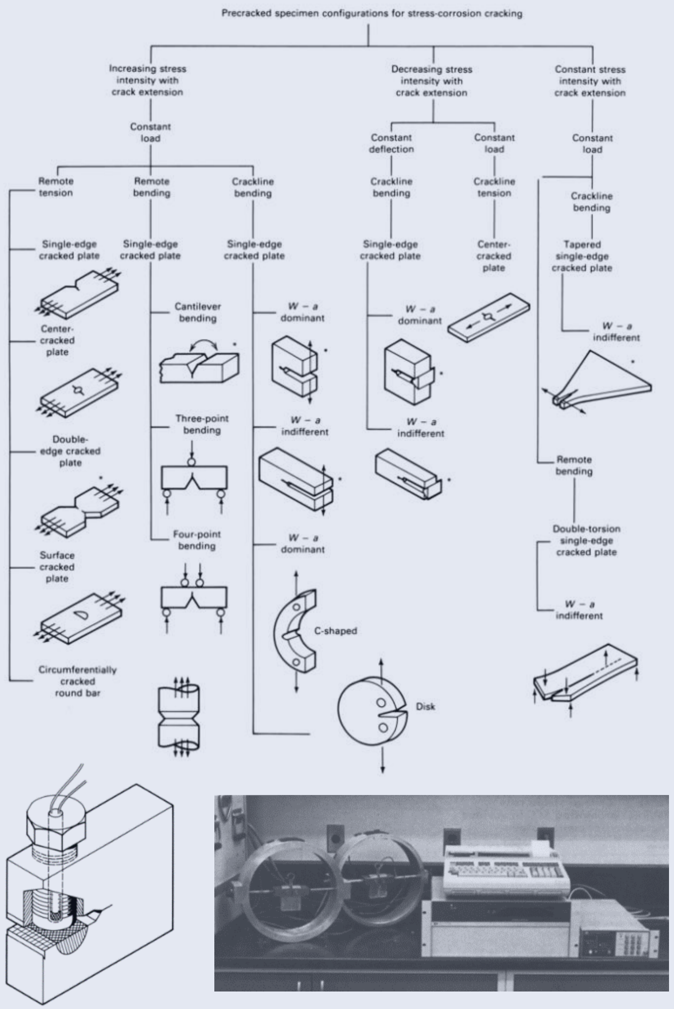
Corrosion, Oxidation, Erosion, SCC/EAC, Other

Simulated Service and Relevant Environment Testing

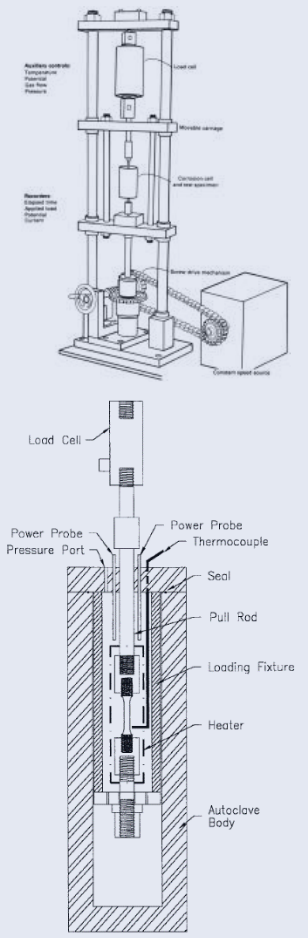
Static Loading of Smooth Specimens



Static Loading of Precracked (Fracture Mechanics) Specimens



Environmental – High Pressure – High Temperature



Typical Material Assessment Approach – New Environment

Phase I:

- Various candidate materials and welds (both SSH and PH):
  - Austenitic Stainless Steels
  - Nickel-Iron-Base Superalloys
  - Cobalt-Base Superalloys
  - Nickel-Base Superalloys
- Smooth C-ring or Bent Beam configuration
- Higher stresses
- Higher temperatures
- Higher thermal cycles per day
- Short exposure (e.g. two weeks)
- Detailed metallurgical evaluation (OM/SEM/EDS/MH)
- Downselect most compatible materials systems for structure and ID possible cladding/coating

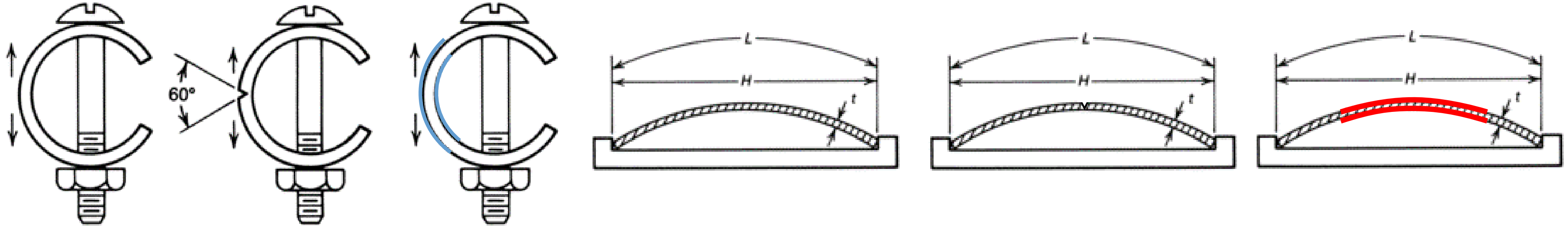
Phase II:

- Downselected material systems:
  - Base material
  - Welded material
  - Coated material
- Smooth and notched C-ring or Bent Beam configuration
- Application stresses
- Application temperatures
- Application thermal cycles
- 30 day exposure
- Detailed metallurgical evaluation (OM/SEM/EDS/MH) + Chemical Analysis
- Downselect final options (hopefully more than one)

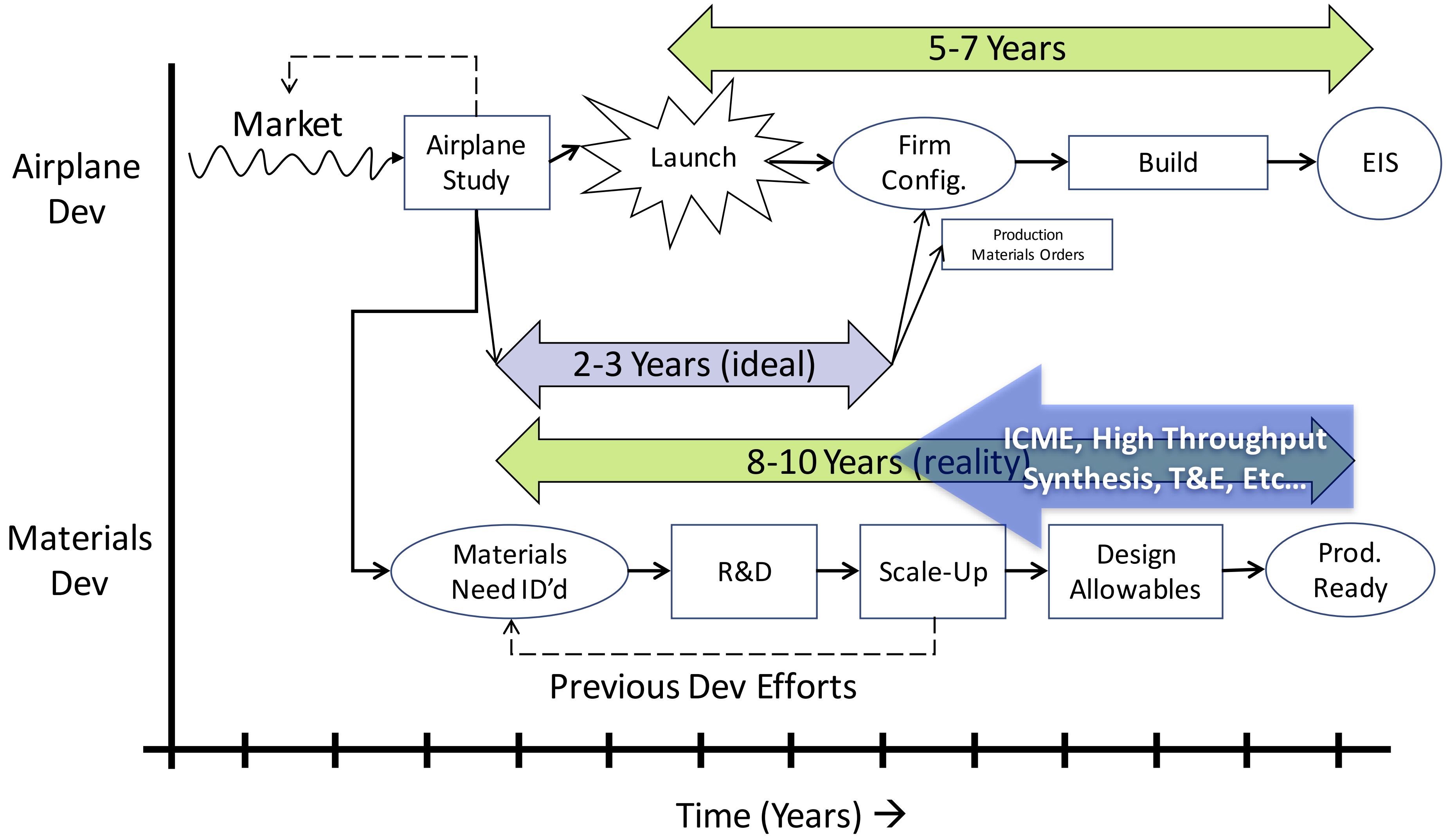
Phase III:

- Bent Beam and Static Tensile and Fracture Mechanics coupon type configurations
- Application stresses
- Application temperatures
- Application thermal cycles
- 90+ day exposure (Bent Beam) and shorter term for other
- Detailed metallurgical evaluation (OM/SEM/EDS/MH)
- Residual mechanical properties (on bent beam samples)

- Subcomponent level testing



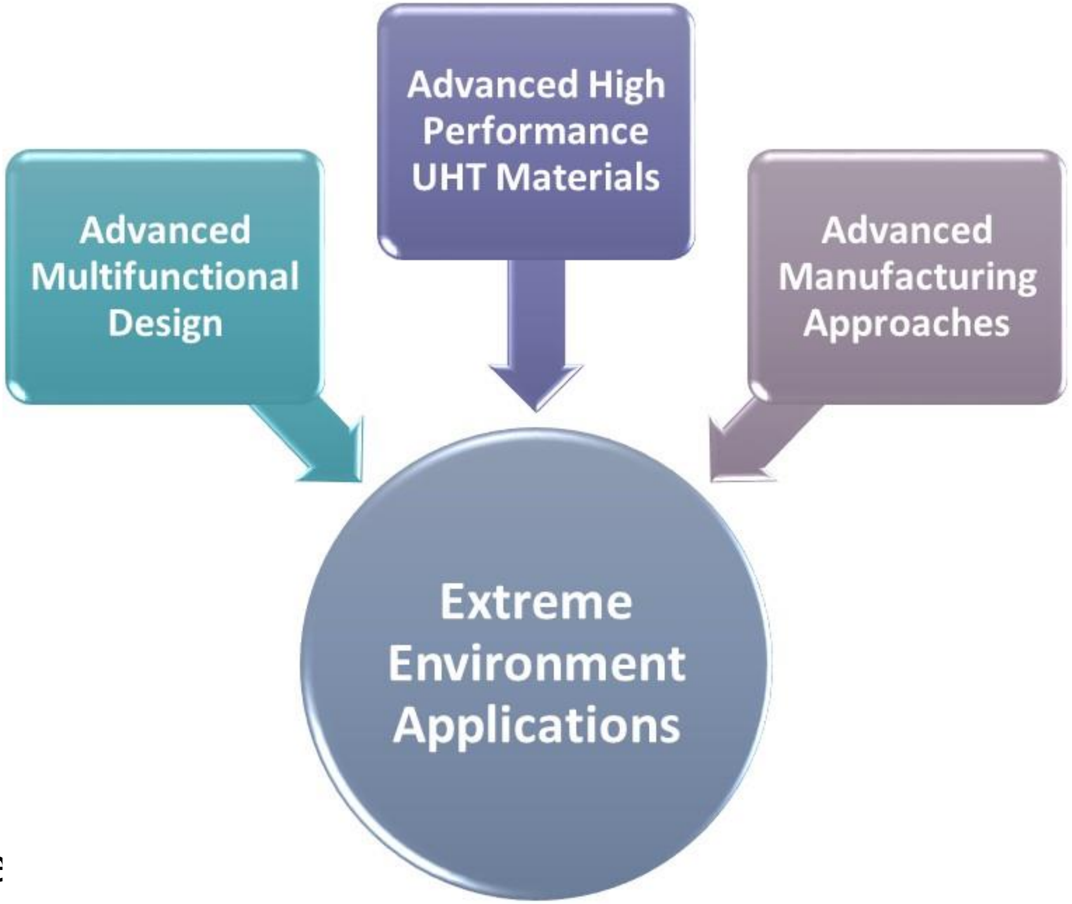
Airplane Development vs. Airplane Material Development

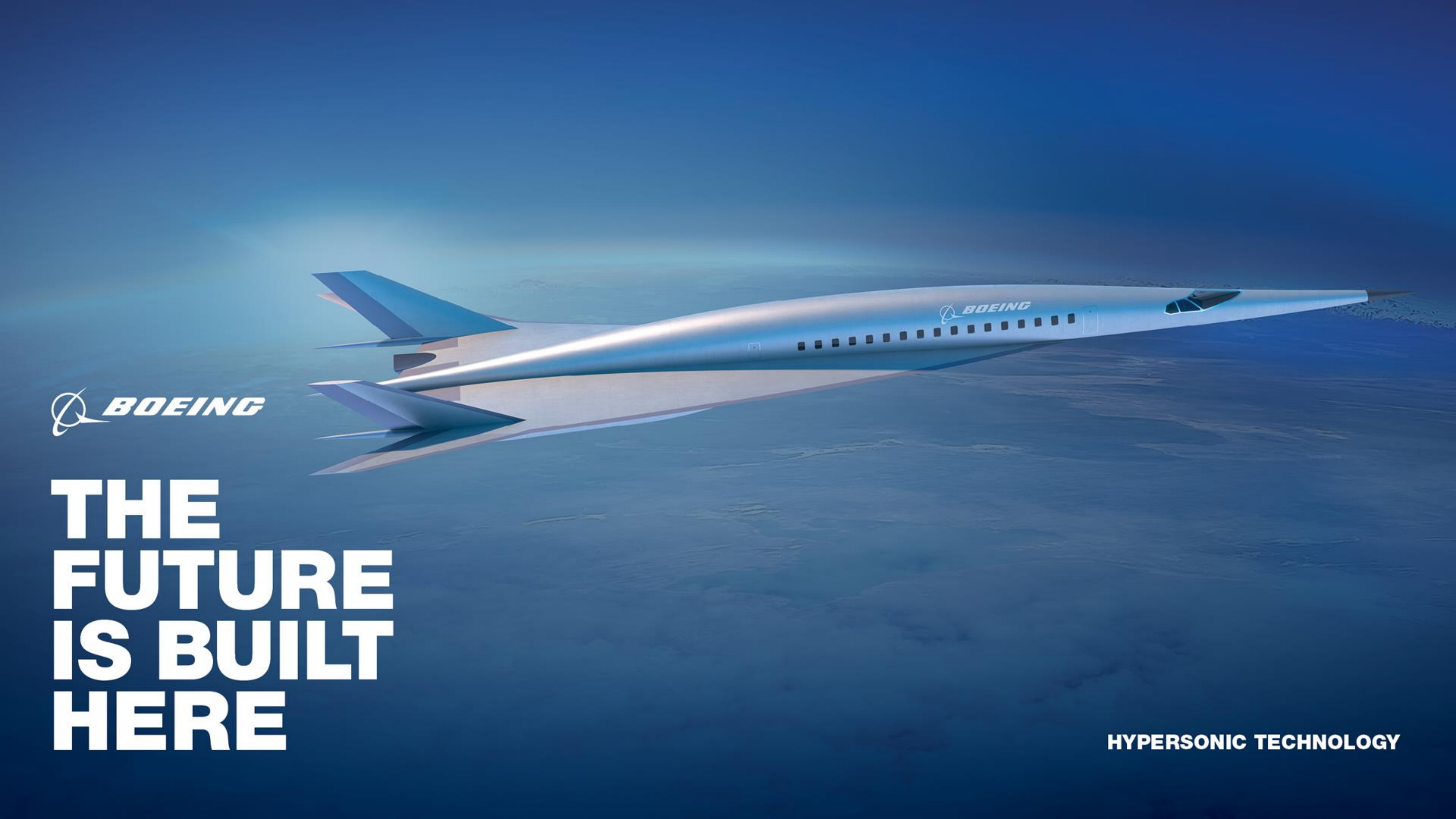


Significant Risk Mitigation is Required to Certify a New Structural Material

Concluding Remarks

- Materials are vital part of aircraft / spacecraft performance improvements
- A new material system must earn its way onto the aircraft:
  - Targeted Application
  - Breakthrough performance improvements
  - Value and affordability across the life cycle
- Significant improvements must be realized to offset development/certification costs
- We need to:
  - Implement high throughput synthesis, testing, and evaluation approaches
  - Get smarter/leaner with R&D activity + team up + leverage resources whenever possible
  - Implement Integrated Computational Materials Engineering as early as possible
  - Choose appropriate application with the best business case
  - Integrate various technologies and incorporate multiple functionality

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